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(54) Title: A PROCESS FOR FRACTIONING CROP INTO INDUSTRIAL RAW MATERIAL

(57) Abstract

The invention relates to a process for fractioning crops by using a combined dry and wet milling and extraction methods, whereby extruding or percussive devices and operating methods are used in the dry milling step, and a thermal treatment, solvent extraction and mechanical treatment, which destroy most of the enzymes, are combined in the first wet milling step.

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A process for fractioning crop into industrial raw material

Besides the use for conventional foodstuff purposes, the botanical or chemical components of crop raw material have been isolated to be used for industrial purposes. The most extensive industrial implements are starch separating processes by using corn, wheat and barley as a raw material. The main by-products of these processes are glutene, which is recoverable from corn and wheat, other protein fractions, corn oil, wheat germ oil as well as fractions mainly used as feed. The methods used in the process can be classed into wet milling methods or combinations of dry process steps and wet milling.

In grain fractioning, various mechanical methods are used, such as screening, air classification, sedimentation as well as extracting and dissolving processes. For oat grinding, milling devices generally suitable for the grinding of other crops have been applied. The US patent specification 4220 287 describes a roll milling device equipped with either smooth or fluted rolls.

For oat fractioning, prior known methods in most cases relate to separating an individual component from the oat grains. The protein separation of oats has been an object for the most extensive study, the central method having been dissolving protein into basic solutions and precipitating by means of acids at the isoelectric point.

In several described processes, the alcalic extraction is preceded by a solvent extraction by means of hydrocarbon, halogenated hydrocarbon, aliphatic alcohols, or aceton, in order to remove the avenol, aiming at facilitating the subsequent separating steps and at improving the preservability of the end products. The impact of various solvents and temperatures on the extraction

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yield is known through scientific literature as well as the GB patent specification 1 526 553, among others. In certain methods, such as the one described in the US patent specification 4 211 801, an organic solvent has been used as a medium to separate bran and endosperm fractions from each other.

The separation of oat gum is described in several processes, as in the method of the US patent specification 4 028 468, as well as in scientific literature, being accomplished by extracting into an aqueous or alcalic solution and by precipitating with the aid of aliphatic alcohol or ammonium sulphate. In some processes, like the one described in the US patent specification 4 435 429, the gum material is allowed to be hydrolyzed in order to reduce the viscosity, and is not recovered.

The antioxidative effects of oats are known from research publications. Phenolic compounds, like coffee and pherulic acid derivatives, are considered major antioxidative factors, however some other oil components, tocopherols, among others, also have a synergetic effect. Nevertheless, this phenomenon has not been technically exploited. In certain processes, as the one described in the GB patent specification 1 526 553 and US patent specification 4 211 801, hydrogen peroxide is used to clarify the oil, and this procedure can be considered to bring about the inactivation of antioxidative factors possibly present. It is likewise known that many other fatty vegetable grains contain polyphenol compounds that have an antioxidative effect. The limited solubility into fats of these compounds has prevented their technical exploitation.

The methods for oat fractioning disclosed in patent literature so far rarely present the final fraction yield, which is one of the most essential criteria when evaluating the profitability of the process. The exact composition of the final fractions is rarely expressed, and most seldom information about the physical or other properties of the end products, which would allow to deduce properties of the fraction significant for industrial use,

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such as the viscosity and the molecular weight with regard to the vegetable gum fraction, the purity or residual protein content of the starch fraction and the proportions of various lipide fractions in the fat fraction. In the cited methods, the concentration of a component to a certain fraction is usually achieved, but not the degree of concentration or purity prerequisited by the technical use of the fraction. Major known oat fractioning processes are disclosed in the introduction of the US patent specification 4 435 429. As far as is known, none of them has resulted in a permanent technical application.

The object of the present invention is a process for fractioning crop grains, in particular oat grains, so as to obtain a high yield and the purity degree required by technical use, for several of the isolated fractions, and to retain as far as possible the functional properties characteristic of the components of the various fractions. This is achieved according to the invention by using extruding or impact devices and operating methods in the dry milling step of the crop, and by combining a heat treatment, solvent extraction and mechanical treatment, which destroy the major part of the enzymes, in the first wet milling step.

Among the functional properties of the components, the purity of the starch is to be considered the most important in particular with regard to the residual protein content, the viscosity properties with regard to the vegetable gum fraction, the anti-oxidative effect and the content of emulsifying components with regard to the fat fraction, and the aminoacid composition with regard to the protein fraction. The possible application fields for these fractions can comprise, as to the starch, food, paper, board, plastic and chemical industry among others, as to the vegetable gum fraction, the use in thickening agents in food, chemical and pharmaceutical industry, and as soluble nutritive fiber in food and pharmaceutical preparations, for the fat fraction, the use in edible fats and in the preparation of emulsifiers, and for the protein faction, the use in foodstuffs and feeds.

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In the first step of the process, the outer covering layer of the oat grains, the husks, are removed by means of a known technique, unless hulless oat species are used. The dehulling should be carried out so as to remove a minimum of the portion below the pericarp layer.

The removal of avenol can be optionally carried out in the different steps. However, an efficient extracting effect requires either milling or flocculation of the grains. For this reason, the first size reducing operation subsequent to the dehulling is preferably accomplished by using a roll mill or some other appropriate device, in which the particle adhesion to the various parts of the device caused by the fat can be prevented by means of the operating method, the sieve dimensioning and possibly also air jets. A suitable procedure for this purpose is e.g. grinding through a smooth pair of rolls and subsequent screening with the aid of a sieve having a 0.2 to 0.5 mm mesh.

The fraction remaining on the sieve can be further reduced by using an impact mill, for instance a hammer mill without sieves. The fraction thus reduced is screened through a 0.1 mm mesh sieve e.g. by a an air jet sieve.

In terms of the present invention, it is important to avoid a cutting operation as far as possible in the size reducing operations, so that the aleurone layer of the grain and the immediately connected portion containing vegetable gum are retained as great particles. The cohesion of these portions is improved by the fat content and a suitable moisture content in the grinding step, and it can be increased by denaturalizing protein e.g. by means of a heating treatment.

According to the final purposes of use of the fractions and to the composition and quality requirements, the extraction of lipides is performed on the entire batch or is concentrated only to the bran portion, where the fat content is higher than in the endo-

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sperm fraction. Preferable solvents according to the invention are polar solvents like 2-propanol, ethanol or aceton, or mixtures of these and water, in order to increase the amount of extracted polar lipides and the total yield of lipides. The preferable temperature range is 50 to 90 C. In order to minimize the oxidation and hydrolyse reactions of the lipides, it is important to inactivate a major part of the lipase and lipoxydase activity of the oat grains by means of a heating or other denaturalizing treatment carried out as early as possible. The same treatment inactivates the beta-glucanase activity and denaturalizes the proteins of the grain. The treatment can be effected e.g. by vaporizing at the flattening stage of the grains by means of prior known technique or by extracting by a hot solvent, whereby the denaturalizing and inactivating effect on the enzymes of the solvents and/or solvent-water systems and/or acidity also can be exploited. However, the heating treatments have to be dimensioned so as to avoid a simultaneous sizing of the starch. The polarity and/or density of the organic solvent is adjustable by adding water or other organic solvents.

The extraction step can be combined with the separation of the endosperm and bran fractions. Provided that the size reducing operations preceding the extraction have been effected as indicated above, the endosperm fraction is separated in the extraction and under the effect of the mechanical forces then exerted as a finely divided fraction and the bran remains in the coarser fraction. These can be separated from each other by screening in a solvent suspension by using a 0.08-0.125 mm mesh sieve or by means of sedimentation or hydrocyclonic treatments.

The most valuable component of the endosperm fraction is the starch. It can be separated from the other components of the endosperm fraction by means of techniques known in starch industry, such as sedimentation or hydrocyclonic treatments or air classification. Besides in the aqueous phase, the sedimentation and

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hydrocyclonic treatments can also be carried out in an organic solvent. When aiming at low residual protein contents or wishing to reduce the number of operation steps, extraction from an alcalic solvent is preferably used to separate the protein from the starch.

When using dry separating operations, the bran fraction remaining on the sieve is approx. 27 to 35% of the amount of dehulled oats. By choosing favourable processing conditions the proportion of the bran fraction can be reduced to ca. 20% of the mass of the dehulled oats by using the separation in a solvent suspension described above. The bran fraction thus concentrated is capable of being used as such in order to increase the nutritive fiber content of foodstuffs and clinical alimentary preparations, but the beta-glucane can be further separated for use as a soluble nutritive fiber or for the preparation of thickening or binding agents.

The beta-glucane is extracted from the bran fraction in a water or alcalic solution, at a temperature of 50 to 75 °C, preferably at a temperature of ca. 70 °C. In order to make it technically possible to separate the viscous extracting solution from the solid matter, the amount of extracting solution must be 200-fold, preferably 300-fold to the amount of beta-glucane in the bran. When using alcalic extraction the protein of the bran is dissolved at the same time. It is precipitated at the isoelectric point and is separated e.g. by centrifugation. The pH of the clear solution is now raised to 7 pH, and the beta-glucane is precipitated by means of alcohol, e.g. ethanol or isopropanol or ammonium sulphate. When precipitating by means of alcohol, the beta-glucane is precipitated as a threadlike deposit easy to separate by screening. The concentration obtained can be further purified by elutriating into a small amount of alcohol. The beta-glucane can be retained suspended in the alcohol without deteriorating the viscosity properties, or it can be dried by air drying or at under-pressure.

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When using polar solvents for extracting the fat it is known that a greater amount of polar lipides are transferred into the extract than by using non-polar solvents. It has been observed in this study that the extraction of compounds having an antioxidative effect is strongly dependent on the polarity of the solvent. It has also been observed that they remain dissolved in avenol, unless it is further refined to remove the polar lipides, and that such an avenol has an antioxidative effect also when being added to other oils.

The invention is further described below by means of examples.

Example 1

Dehulled oats were ground through a smooth pair of rolls and screened through a 0.2-0.3 mm mesh cylindrical sieve. The weight part of the separated fine endosperm fraction was 60-65%, its starch content was 80-81%, protein content 10-115, fat content 5.8% and beta-glucane content ca. 1%. Correspondingly, the starch, protein, fat and beta-glucane contents of the bran fraction were respectively 40-43; 18-20; 7,8 and 11-12%. The nutritive fibre content of the bran fraction was 29%.

Example 2

The dehulled oats were ground through a smooth pair of rolls, treated by means of a hammer mill without screen, the mixture was suspended into a sixfold weight quantity of water-2-propanol mixture (15:85% v/v), and the mixture was extracted in this suspension for 2 hours under agitation. By screening through a 0.125 mm mesh sieve a fine endosperm fraction was separated from the suspension, the dry weight of which was 71% of the weight of the dehulled oats. Its starch, protein, fat and beta-glucane contents were respectively 85; 10.5; 1.5 and 1%. Correspondingly, the weight part of the bran fraction remaining on the sieve was 24% of the amount of dehulled oats and its starch, protein, fat, beta-glucane contents were 35; 23; 2 and 15.5%, its ash content 3.5% and nutritive fiber content 35%.

Example 3

The bran fraction of example 1 was concentrated by elutriating into a sixfold weight amount of water-alcohol mixture at 75-78 °C which contained 80 volume-% of 2-propanol and 20 volume-% of water. The mixture was vigorously homogenized by a blade mixer during approx. 3 minutes, and was subsequently mixed by a paddle mixer during 1 to 2 hours. The solid matter was screened by a 0.125 mm mesh sieve, whereby the concentrated bran fraction remained on the sieve, the dry weight of which was approx. 20% of the weight of the dehulled oats batch. Its beta-glucane content was 17 to 18% , the total nutritive fiber content 35 to 39% and protein content 20 to 24%. The starch content of the solid matter having passed through the screen was 83 to 86%. When effecting a batch process, ca. 75% of the oil content of the bran was transferred into the extraction solution by one extraction.

Example 4

The bran concentration prepared according to example 3 was extracted to a 9.5 pH value in a regulated sodium hydroxide solution at a temperature of 70 °C during 2 hours. In order to facilitate the further processing of the viscous solution subsequent to the extraction the amount of extracting solution was ca. 40-fold to the amount of solid matter, i.e. 200 to 300-fold to the amount of beta-glucane. The extracted protein was precipitated by adjusting the pH vaue of the solution to pH 4.5, and the protein was separated by centrifugation. The pH of the solution was now adjusted to 7.0 and the beta-glucane was precipitated by directing the viscous beta-glucane solution into ethanol or propanol, under simultaneous vigorous agitation, until the alcoholic concentration of the mixture was reduced to 50% by weight. The beta-glucane was precipitated as a thread-like mass which was easily separated from the solution onto a 0.2 mm mesh sieve or by means of a filter press. After washing with 2-propanol the deposit was dried by evaporating the solvent at a temperature of 35 °C. The beta-

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glucane content of the preparation was 80%, the residual protein content was 2 to 4% and the starch content was 2 to 3%.

Example 5

The starch was separated from the endosperm fraction of example 2 by extracting the endosperm fraction by a sodium hydroxide solution, the pH of which was 9.5, at a temperature of 40 °C. The solid matter was separated from the solution by means of a hydrocyclone. The residual protein content of the starch obtained after repeated washing steps was 0,4% and the residual fat content was 0.5 to 0,5%.

Example 6

The solvent was removed by evaporation from the isopropanol extract of example 2. The oil obtained was added to refined corn oil 3%, and the stability of the oil to oxidation was tested by the Rancimat procedure and device by leading oxygen through the oil at 110 °C, by absorbing the oxidation results produced by the exhaust gas into an absorption solution and by observing the change taking place in the electroconductivity of the latter. The increase of the oxidation induction time caused by the avenol addition was the same as when 0.2% butylated hydroxyanisol was added. The antioxidative properties were observed also in the lecithin fraction of the oil.

Example 7

1000 kg of dehulled and steam treated oat kernels were ground with a pilot-scale roll milling equipment. From the rolls, the powder was conducted to a 670 µm vibrating screen, and the remaining bran was further purified by a bran centrifugator. The proportion of slate bran was 41% by weight and the beta-glucane content 9.0%, the proportion of flour was 50% by weight and its glucane content 1.6%.

By further screening of the bran with the aid of an air jet sieve the beta-glucane content of the bran remaining on a 75 µm sieve was increased to 11.7%.

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The grinding of the oat flakes dehulled, flattened and solvent extracted from the same raw material was technically difficult to carry out with the equipment. Owing to the extraction of the lipides, the flakes broke and the share of bran after the screening was ca. 19% by weight and the beta-glucane content 11% and the beta-glucane content of the endosperm fraction having penetrated was 2.6%. The beta-glucane content of the bran could not be further concentrated by means of the air jet sieve.

The above examples are based on experiments carried out with oat grains. Besides oat, the process is suitable, where applicable, also for fractioning the grains of other crops.

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Claims

1. A process for fractioning crops, in particular oats, into industrially or nutritionally usable fractions, by using dry milling or a combined dry and wet milling and extraction methods, characterized in that prior to the dry milling step, the protein is denaturalized by a heat and/or solvent treatment, extruding and/or percussive, but not cutting, grinding methods are used in one or several steps for the grinding, and/or for extracting lipides, polar solvents are used at a temperature of 50 to 90 °C, and/or a base extraction is carried out at a temperature of 50 to 75 °C in order to dissolve the proteins and/or beta-glucanes.

2. A process according to claim 1, characterized in that the bran is concentrated to a high fiber and/or protein content without separating protein and/or beta-glucane.

3. A process according to claim 1 or 2, characterized in that the fractioning is carried out without including extraction of the lipides by organic solvents.

4. A process according to claim 1, 2 or 3, characterized in that the separation of the bran fraction from the endosperm fraction is carried out by grinding, screening, centrifugating or by hydrocyclones in an organic solvent.

5. A process according to claim 1, 2 or 4, characterized in that the polarity and/or density of the organic solvent is adjusted by adding water or other organic solvents.

6. A process according to claims 1, 3, 4 or 5, characterized in that after precipitating the beta-glucane, it is kept suspended in an organic solvent before drying.

BNSDOCID: <WO___8901294A1_I_>

7. A process according to claims 1, 2, 4, 5 or 6, characterized in that in order to retain the antioxidative properties the lipide fraction is kept unrefined, or when fractioning or refining further, the solubility of the antioxidative components is ensured by keeping the proportion of polar solvents or lipides sufficiently high.

BNSDOCID: <WO___8901294A1_I_>

AMENDED CLAIMS

[received by the International Bureau on 11 January 1989 (11.01.89)
original claims 1-4 unchanged; claim 5 added;
original claims 5-7 renumbered 6-8 (2 pages)]

1. A process for fractioning crops, in particular oats, into industrially or nutritionally usable fractions, by using dry milling or a combined dry and wet milling and extraction methods, characterized in that prior to the dry milling step, the protein is denaturalized by a heat and/or solvent treatment, extruding and/or percussive, but not cutting, grinding methods are used in one or several steps for the grinding, and/or for extracting lipides, polar solvents are used at a temperature of 50 to 90 °C, and/or a base extraction is carried out at a temperature of 50 to 75 °C in order to dissolve the proteins and/or beta-glucanes.

2. A process according to claim 1, characterized in that the bran is concentrated to a high fiber and/or protein content without separating protein and/or beta-glucane.

3. A process according to claim 1 or 2, characterized in that the fractioning is carried out without including extraction of the lipides by organic solvents.

4. A process according to claim 1, 2 or 3, characterized in that the separation of the bran fraction from the endosperm fraction is carried out by grinding, screening, centrifugating or by hydrocyclones in an organic solvent.

5. A process according to claim 1, 2 or 3, characterized in that the separation of the bran fraction from the endosperm fraction is partly or wholly carried out by grinding, screening, centrifugating or by hydrocyclones in cold water.

6. A process according to claim 1, 2 or 4, characterized in that the polarity and/or density of the organic solvent is adjusted by adding water or other organic solvents.

BNSDOCID: <WO___8901294A1_I_>

7. A process according to claims 1, 3, 4 or 5, characterized in that after precipitating the beta-glucane, it is kept suspended in an organic solvent before drying.

8. A process according to claims 1, 2, 4, 5 or 6, characterized in that in order to retain the antioxidative properties the lipide fraction is kept unrefined, or when fractioning or refining further, the solubility of the antioxidative components is ensured by keeping the proportion of polar solvents or lipides sufficiently high.

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INTERNATIONAL SEARCH REPORT

International Application No PCT/FI88/00125

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all) [6]

According to International Patent Classification (IPC) or to both National Classification and IPC [4]

A 23 J 1/12

II. FIELDS SEARCHED

Minimum Documentation Searched [7]

Classification System	Classification Symbols
IPC	A 23 J 1/00,/12; A 23 L 1/10
US Cl	426:18,44,436,615,618,656

Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched [8]

SE, NO, DK, FI classes as above.

III. DOCUMENTS CONSIDERED TO BE RELEVANT [9]

Category [10]	Citation of Document, [11] with indication, where appropriate, of the relevant passages [12]	Relevant to Claim No. [13]
X	WO, A1, 86/01080 (BARCO, INC) 27 February 1986 see claim 1 and fig & EP, 0192677 JP,T,61502940	1-7
X	US, A, 4 028 468 (HOHNER et al) 7 June1977 see example & CA, 1085384	1-7
Y	US, A, 4 211 801 (RICHARD OUGHTON) 8 July 1980 see example 1 & FR, 2314672 DE, 2627137 GB, 1527101 AU, 496745 JP, 52001048 CA, 1087451 SE, 7606863	1-7

.../...

* Special categories of cited documents: [10]

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IV. CERTIFICATION

Date of the Actual Completion of the International Search	Date of Mailing of this International Search Report
1988-10-04	1988 -11- 11
International Searching Authority	**Signature of Authorized Officer**
Swedish Patent Office	Kerstin Birgit Jonsson

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BNSDOCID: <WO___8901294A1_I_>

III. DOCUMENTS CONSIDERED TO BE RELEVANT (CONTINUED FROM THE SECOND SHEET)

Category *	Citation of Document, with indication, where appropriate, of the relevant passages	Relevant to Claim No
Y	EP, A1, 0 028 518 (DU PONT CANADA INC.) 13 May 1981 see claim 1 & JP, 56081104 CA, 1133446	1-7
Y	US, A, 4 211 695 (RICHARD OUGHTON) 8 July 1980 & DE, 2657034 DE, 2657103 FR, 2351605 FR, 2351606 FR, 2351704 FR, 2361071 FR, 2361156 US, 4154728 GB, 1552012 JP, 52073899 JP, 52073900 AU, 505480 AU, 505766 CA, 1080700 CA, 1087611 CA, 1095897 SE, 7613741 SE, 7613742	1-7
A	US, A, 3 909 288 (POWELL et al) 30 September 1975	1-7
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BNSDOCID: <WO___8901294A1_I_>